MBE growth of (In)GaAsN on GaAs using a constricted DC plasma source

A E Zhukov[1]**, R Zhao**[2]**, P Specht**[2]**, V M Ustinov**[1]**, A Anders**[3] **and E R Weber**[2]

[1] Ioffe Physico-Technical Institute Russian Academy of Sciences, 26 Politekhnicheskaya, St Petersburg, 194021, Russia
[2] Department of Materials Science and Mineral Engineering University of California at Berkeley, Berkeley, California 94720-1760, USA
[3] Lawrence Berkeley National Laboratories, Berkeley, California 94720, USA

Abstract
(In)GaAsN epilayers were grown on GaAs substrates by molecular beam epitaxy (MBE) with a DC constricted-plasma source. Nitrogen incorporation efficiency, crystalline quality, surface morphology and luminescent properties of the epilayers were studied and correlated with various operation regimes of the source. The nitrogen incorporation efficiency grows with increasing plasma discharge current. The maximum nitrogen concentration in the epilayer is as high as 3.7 %. GaAsN as thick as 0.35 μm can be pseudomorphically grown on GaAs, and photoluminescence is observed at room temperature. The crystalline quality and surface morphology of an epilayer can also be improved by reducing the ion damage at low plasma discharge current. The DC constricted-plasma source is a promising alternative nitrogen plasma source for investigating InGaAsN materials.

1. Introduction

The (In)GaAsN/GaAs system is now considered a promising candidate for application in long-wavelength lasers on GaAs substrates [1, 2]. Edge-emitting quantum well diode lasers [3] and VCSELs [4] operating at 1.3 μm have recently been successfully fabricated. InGaAsN quantum well and quantum dot heterostructures emitting at room temperature in the wavelength range as long as 1.43 and 1.52 μm, respectively, have been demonstrated [5, 6]. It is also worth mentioning an interesting alternative to InGaAsN for developing long-wavelength laser sources on GaAs substrates: a GaAsSbN quantum well was shown to exhibit a room-temperature photoluminescence peak wavelength as long as 1.52 μm [7]. The active nitrogen species in MBE growth of (In)GaAsN are usually provided by radio-frequency (rf) nitrogen plasma sources having low efficiency and also causing ion damage to the films. Therefore, it is potentially useful to introduce and study an alternative nitrogen source suitable for MBE growth of high-quality N-containing heterostructures.

Only scarce information is available concerning the MBE growth of (In)GaAsN structures with DC plasma source and their characterization [8]. However, these and our initial experiments indicate that the DC plasma source potentially offers high efficiency of nitrogen incorporation, high growth

stability and reproducibility. Moreover, the DC plasma source is much less expensive. The present work is concerned with the MBE growth of GaAsN and InGaAsN epilayers on GaAs (001) substrates, using a novel type of nitrogen plasma source: DC constricted plasma source (CPS) [9, 10]. The CPS is characterized by low density and energy of ions. It has been successfully used to grow high quality GaN epilayers [9]. Here we introduce this plasma source to the InGaAsN community. The nitrogen incorporation efficiency, crystalline quality, surface morphology and photoluminescence (PL) properties of the epilayers are presented and correlated with various operation regimes of the nitrogen plasma source.

2. Experiment

The samples were grown in an Intevac Gen II MBE machine equipped with solid sources of Ga, In and As, ion- and cryo-pumps, as well as a standard reflection high-energy electron diffraction (RHEED) system. Vertical gradient-freeze grown (VGF) and epi-ready semi-insulating GaAs(100) substrates (AXT, Fremont, CA) were used. First, a 0.1 μm thick GaAs buffer layer was grown at 580 °C, which was followed by 5 min growth interruption to reduce the substrate temperature to 450 °C and to ignite and stabilize the nitrogen plasma.

Then a 0.35 μm thick (In)GaAsN layer was deposited and covered with a 20 nm thick GaAs capping layer. The growth rate was 1 monolayer per second (ML); the equivalent pressure of the arsenic beam was 2×10^{-5} Torr (As-rich conditions). The plasma source was positioned 8 inches away from the substrate to get a uniform nitrogen beam flux and avoid the shadowing effect. The operating regimes of the plasma source for MBE growth of N-containing epilayers are discussed below. In all the employed growth regimes, the background pressure was kept as low as 10^{-5} Torr. A Siemens D5000 x-ray diffraction system was used for crystal characterization. Photoluminescence (PL) was excited by an Ar$^+$ laser (514.5 nm, 50 W cm^{-2}) and detected with a cooled Ge photodiode.

The DC constricted-plasma source (CPS) is based on a special kind of DC glow discharge. It is characterized by a small orifice (constriction, about 1 mm in diameter) separating a small discharge chamber from the MBE growth chamber. The discharge current is concentrated at the constriction, leading to ionization greater than that found in traditional glow discharges. Additionally, the constriction serves to provide low pressure in the growth chamber and a directed flow of plasma and activated neutral species. The plasma flow velocity is several times 100 m s^{-1} (Mach number >1) and depends on the gas flow rate. Although the degree of ionization is low, i.e., the density of ions is much lower than the density of the neutral nitrogen species, the CPS is effective for film growth since the flow contains a variety of nitrogen species. They include excited and ionized molecules and nitrogen atoms in the ground, excited or ionized state. All of the mentioned species carry internal energy, as opposed to kinetic energy, and thus can be incorporated into the growing film [10].

The control parameters of the CPS, which can affect the flux of the active species and the nitrogen content in the epilayer, are the nitrogen flow rate and the plasma discharge current. However, their effects on the nitrogen incorporation efficiency and the minimum and maximum possible nitrogen concentration in the epilayer are to be evaluated, which is among the goals of this work. A 5 sccm Tylan (Millipore) FC-2950M series flow control system and a Glassman ER series DC power supply (100 mA, 3 kV) were used.

3. Results and discussion

3.1. Plasma analysis

We first studied the range of possible variation of the operation parameters: gas flow rate F and plasma discharge current I. Their minimum values are limited by the physical processes in the ignition chamber of the plasma source, the maximum values of I and F being restricted by the experimental equipment (100 mA and 5 sccm, respectively). For a given flow rate we obtained the minimum current, I_{min}, at which the plasma discharge was maintained. Figure 1 shows the dependence of I_{min} on F. It can be seen that I_{min} decreases steeply with F at low flow rates (0.2–0.8 sccm), being practically unchanged at higher F (1–5 sccm).

Also, an additional current-related critical parameter was found, characterizing the temporal stability of the plasma source. If the operating current is less than a certain flow rate-dependent value, I_S, the plasma discharge will suddenly cease
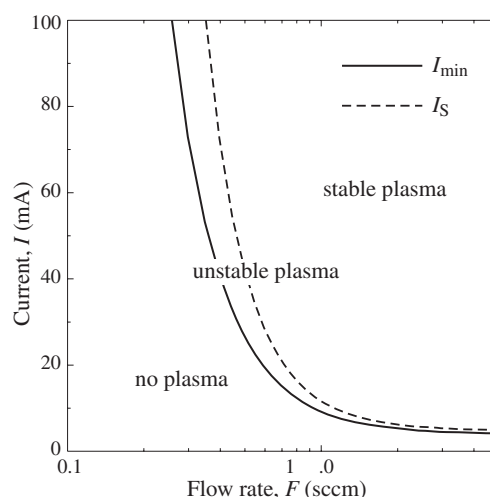

Figure 1. Minimum current maintaining the plasma discharge, I_{min}, (full curve) and the minimum stable discharge current I_S (broken curve) versus the nitrogen flow rate F.

within the first few minutes of operation of the source. The $I_S - F$ dependence is also represented in figure 1 by a broken curve. This dependence is qualitatively very similar to that of I_{min}. However, the difference between I_S and I_{min}, i.e., the range of temporal instability, becomes wider with decreasing flow rate. We believe that random fluctuations of the flow rate around the setpoint value play a crucial role. Even if seldom, a large sudden decrease in the flow rate can extinguish the plasma discharge if the flow rate becomes momentarily lower than the minimum flow rate for a given current. It is obvious that a current in the $I_{min} - I_S$ range can hardly be used to grow bulk materials. However, such a regime can probably be applied to QW growth.

Plasma emission spectroscopy is a powerful tool for characterizing the efficiency of plasma activation [11]. We used an EG&G PARC optical multichannel analyser (OMA III series, model 1460) to take plasma emission spectra from the operating zone of the CPS in the wavelength range 200–800 nm. The optical signal was introduced into the measuring system through a viewport of the MBE chamber, a collimating lens, and an optical fibre. Figure 2 shows the evolution of the emission spectra at $F = 5$ sccm and different plasma discharge currents. Each spectrum consists of several bands corresponding to the first and the second positive series transitions of the neutral N$_2$ molecule and the first negative series of the N$_2^+$ ion (see labels in figure 2). Surprisingly, no strong atomic line at 745 nm is observed in the emission spectra. It has been shown recently that the nitrogen composition in GaAsN layers grown by chemical beam epitaxy (CBE) correlates well with the intensity of atomic lines in the optical spectra of the rf nitrogen plasma [11]. However, to date, there is no clarification of the question as to whether or not excited N$_2$ molecules can contribute to nitrogen incorporation. A characteristic feature of our CPS is that the intensities of all the emission lines grow linearly with increasing plasma discharge current. This behaviour is illustrated in the inset of figure 2, where relative intensities of different spectral bands are shown. It is clearly seen that the relative intensity, i.e., the ratio of the integrated to total light intensity is nearly constant
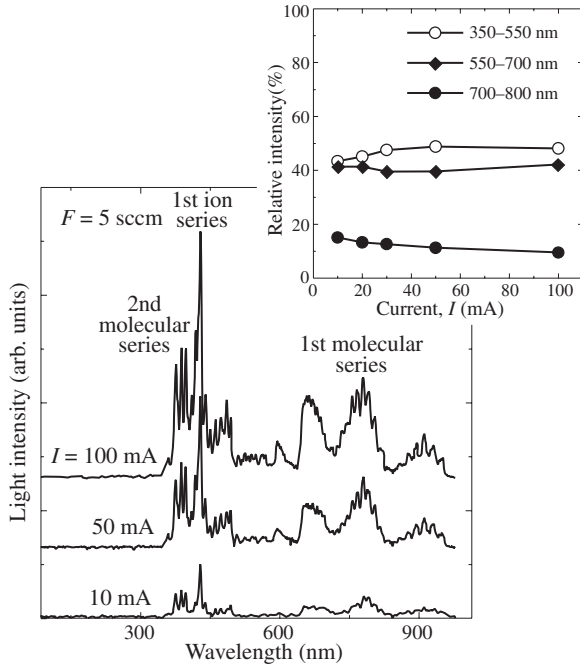
Figure 2. Emission spectra of nitrogen plasma at $F = 5$ sccm and different discharge currents $I = 10, 50,$ and 100 mA. The inset shows relative intensities in several spectral ranges.

with respect to the discharge current. We can assume that the intensity of the specific spectral line(s) associated with the active species is also proportional to the total integrated intensity in the spectral range in question (200–800 nm). Thus, we can use the total integrated optical intensity as a characteristic of the nitrogen plasma activation.

Figure 3 shows the experimental light intensity as a function of the flow rate and the current. The light intensity (or the concentration of the active species, as we have assumed) strongly depends on both the input parameters. In general, it grows when either the discharge current or the nitrogen flow rate increases. However, the light intensity tends to saturate with F, especially at small currents (less than 20 mA). The light–current dependence is also sublinear. We can also observe optical emission even at the minimum stable discharge current, I_S (open spheres in figure 3). This suggests that there must be a minimum nitrogen content that can be reproducibly achieved in a bulk GaAsN epilayer grown using this type of nitrogen plasma source. Further studies of the optical emission in a broad spectral range are desirable, especially those of the spectra taken from the near-substrate region.

3.2. Nitrogen incorporation efficiency

We grew a set of samples, varying the plasma discharge current and the nitrogen flow rate to evaluate the nitrogen incorporation efficiency as a function of the CPS operating parameters. The nitrogen composition and the crystalline quality were evaluated by x-ray diffraction (XRD) rocking curves taken around GaAs(004) (Bragg angle $\Theta_{Br} = 33.0124°$). Figure 4 shows representative (004) x-ray diffraction profiles of the samples grown with different sets of F and I. The x-ray diffraction peak shifts to higher angles when either the current or the flow rate increases. This is in agreement with the above
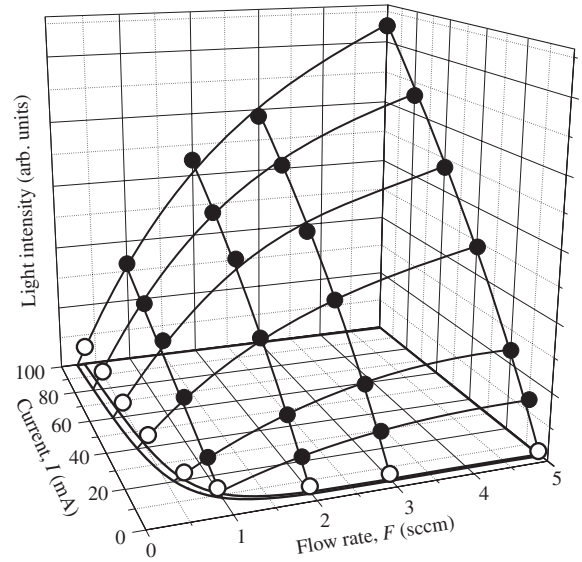

Figure 3. Integrated intensity of light (200–800 nm) emitted by nitrogen plasma as a function of the nitrogen flow rate F and the discharge current I. Open spheres correspond to the light intensity at $I = I_S$.
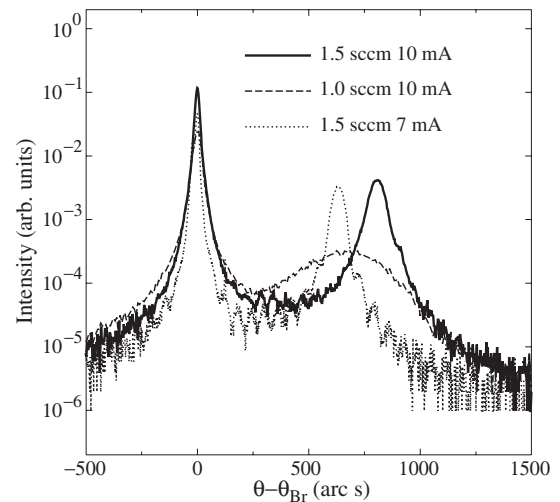

Figure 4. (004) x-ray diffraction rocking curves taken from 0.35 μm thick GaAsN epilayers grown at different discharge currents and flow rates.

results of plasma spectroscopy, which revealed an increase in the light intensity with increasing I or F.

The angle separation between the (004) XRD peaks of the substrate and the epitaxial layer enables us to calculate the nitrogen composition if the in-plane strain is known. Assuming the pseudomorphic growth mode, the nitrogen composition of the samples presented in figure 4 was calculated to be in the range 1.2–1.5 %. A simple estimate using the Matthews and Blakeslee model of mechanical equilibrium [12] gives a critical layer thickness of 98–130 nm for this composition range. This is well below the thickness of our GaAsN layers, equal to 0.35 μm. Thus, partial or full strain relaxation may have to be taken into account.

Strain relaxation can be measured using asymmetric (224) reflection scans. The lattice mismatch in the growth direction,
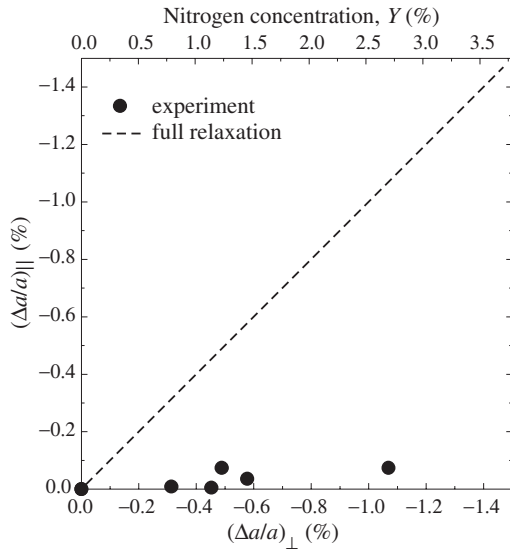
Figure 5. Relationship between lattice mismatch, evaluated from asymmetric (224) x-ray diffraction measurements in the growth direction, $(\Delta a/a)_\perp$, and in-plane, $(\Delta a/a)_\parallel$, for GaAsN epilayers. Broken curve corresponds to the case of full relaxation.

$(\Delta a/a)_\perp$, and the in-plane lattice mismatch, $(\Delta a/a)_\parallel$, were calculated from the peak separation between the substrate and the epitaxial layer in $(224)^+$ and $(224)^-$ reflections. Full circles in figure 5 represent the experimental values of the in-plane lattice mismatch $(\Delta a/a)_\parallel$ as a function of $(\Delta a/a)_\perp$. The dotted curve corresponds to the case of full strain relaxation. The experimental value of $(\Delta a/a)_\parallel$ remains surprisingly small for the whole data set. Even at $(\Delta a/a)_\perp$ as large as -1.1 % (nitrogen content of around 2.7 %), the in-plane lattice constant of the grown GaAsN layer differs from that of the GaAs substrate by less than 0.1 %. These data suggest a pseudomorphic growth mode in spite of the high nitrogen concentration in our samples. Similar experimental results have recently been reported for MOCVD-grown GaAsN epilayers [13]. At present, the reason for the impeded formation and propagation of misfit dislocations in thick GaAsN epilayers is still unknown. This issue needs further investigation by transmission electron microscopy.

Figure 6 shows a three-dimensional plot of the nitrogen concentration Y as a function of the flow rate F and the current I. The nitrogen concentration is a sublinear function of both the discharge current and the flow rate. This dependence is very similar to that of the light intensity (figure 3). This indicates a potential application of the plasma emission spectroscopy to correlate the nitrogen incorporation with the operation parameters of the source. It is likely that, in the future, it will be possible to predict the nitrogen composition using the measured emission intensity.

It should be noted that a minimum nitrogen concentration was observed for samples grown at the minimum stable current I_S for a given flow rate (full curve in figure 6). With a high nitrogen content not always being desirable, we studied in more detail the minimum nitrogen concentration which can be achieved in (In)GaAsN layers grown with the CPS. Figure 6 demonstrates the importance of the discharge current. One clearly observes a distinct decrease in the nitrogen concentration with decreasing discharge current (I_S), although
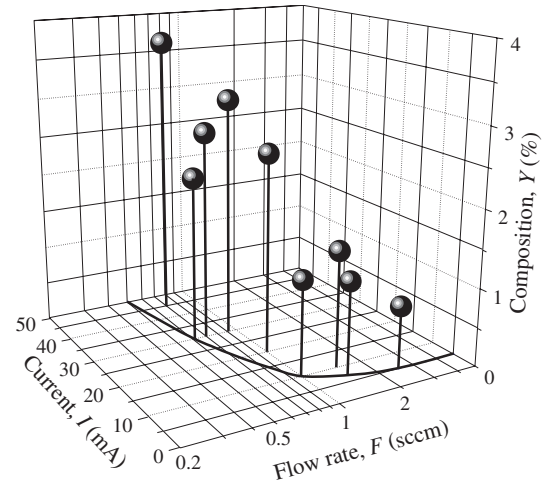

Figure 6. Nitrogen content Y in GaAsN epilayers grown at different discharge currents I and nitrogen flow rates F. The full curve corresponds to the I_S versus F dependence.

the flow rate increases by almost an order of magnitude. The minimum nitrogen composition of 0.84 % was achieved in the sample grown at a flow rate of 3.5 sccm and discharge current as low as 7 mA. At the same time, the CPS provides a sufficient efficiency for nitrogen to be incorporated in a concentration as high as 3.7 % at $I = 45$ mA and $F = 0.7$ sccm. Thus, we can conclude that the plasma discharge current is a more sensitive parameter than the flow rate for this type of nitrogen plasma source.

The flow rate of 1 sccm corresponds to a flux of 9.27×10^{17} nitrogen atoms per second, counting one N_2 molecule as two atoms. Being spread over a 2π solid angle, it provides a flux density, f_N, of 3.69×10^{14} atoms cm^{-2} s^{-1} at a distance of 20 cm from the CPS aperture to the substrate. At the same time, 1 ML of GaAs growth rate is equivalent to $f_{Ga} = 6.26 \times 10^{14}$ Ga atoms cm^{-2}s^{-1}. The efficiency of nitrogen incorporation, η_{in} is the product of the nitrogen activation efficiency of the plasma source, η_{ac}, and the growth condition-dependent sticking coefficient of the active nitrogen species, η_{st}. The nitrogen concentration in the epilayer, Y, is given by

$$Y = \eta_{ac}\eta_{st} \frac{f_N}{f_{Ga}}. \qquad (1)$$

Under the assumption of 100 % activation and 100 % sticking coefficient, 1 sccm nitrogen flow rate will provide a nitrogen concentration in the epilayer as high as 57 %:

$$Y_{max} = \frac{f_N}{f_{Ga}}. \qquad (2)$$

However, the experimental results are 1.22 and 2.56 % at a current of 10 and 20 mA, respectively (figure 6). Combining equations (1) and (2) with the experimental data, we calculated the incorporation efficiency as a function of the discharge current, as shown in figure 7. The incorporation efficiency is a linear function of the discharge current. It should be noted that the variation of the flow rate affects η_{in} only marginally.

Because all the samples were grown under nominally the same growth conditions, which include the substrate temperature, growth rate and As overpressure, we can assume
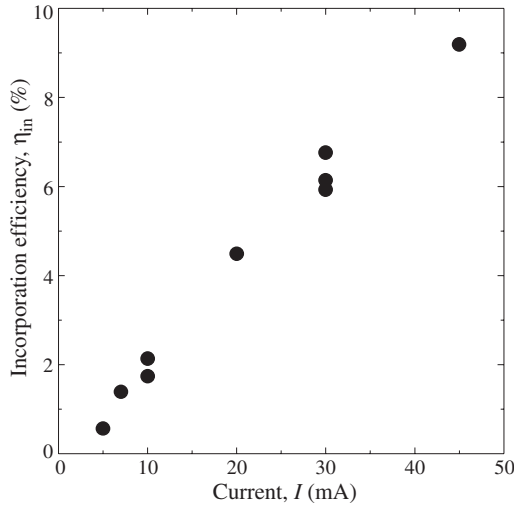
Figure 7. Nitrogen incorporation efficiency η_{in} versus the discharge current I.

that the sticking coefficient is constant. Therefore, the discharge current dependence of the incorporation efficiency also reflects the changes in the nitrogen activation efficiency with respect to the discharge current. The fact that the activation efficiency is mostly governed by the discharge current seems to be very natural, because the current is directly proportional to the flux density of charged particles (electrons and ions), which can generate active nitrogen species through collision processes.

3.3. Structural properties

The data presented in figure 6 show that different sets of the operating parameters F and I give the same nitrogen composition. For example, 7 mA–1.5 sccm and 10 mA–1 sccm correspond to Y of around 1.2 %, as it is also illustrated in figure 4. However, the crystalline qualities of these two GaAsN layers are quite different. While the (004) rocking curve peak of the epilayer from the 10 mA–1 sccm sample is very broad (about 400 s of arc), it is much smaller for the 7 mA–1.5 sccm sample (about 60 s of arc). We found that, generally, a higher flow rate is beneficial for the linewidth of the x-ray diffraction peak. Even for the sample with the highest nitrogen concentration in figure 4 (1.5 %: the 10 mA–1.5 sccm sample) the linewidth remains comparably narrow (about 100 s of arc). It is worth mentioning that the ion flux from the CPS decreases with increasing flow rate at the same discharge current [9]. Thus, the sample surface suffers lesser ion bombardment during growth at larger nitrogen flow rate and the crystalline quality of the epilayer can be improved. Another possible reason for the broad x-ray rocking curves is the nitrogen composition modulation caused by flow fluctuations resulting from the strong $Y-F$ dependence in the low-flow regime. However, further study by TEM is necessary to confirm the last statement.

We also studied the surface morphology of the samples by analysing RHEED patterns and by means of Nomarski microscopy. Figure 8 shows a map of the surface morphology in relation to the operating parameters of the nitrogen plasma source. We found a close correlation between the discharge
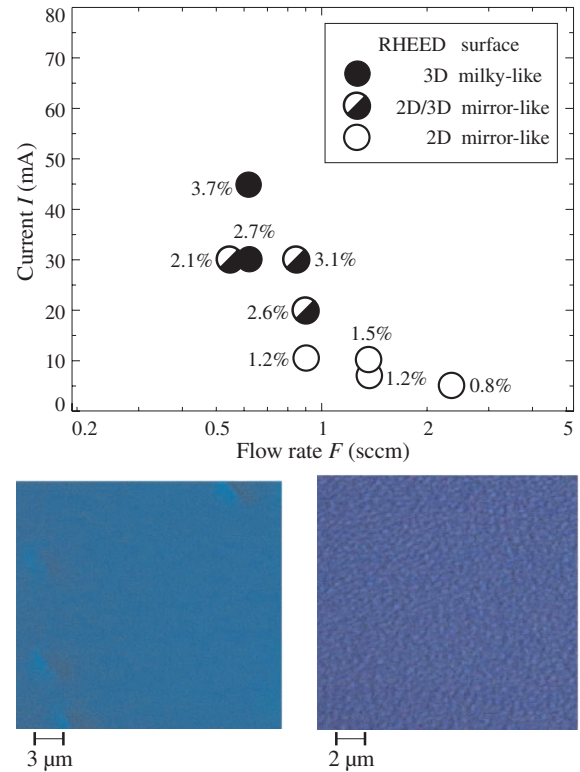

Figure 8. Surface morphologies and RHEED patterns of GaAsN epilayers grown at different discharge currents I and nitrogen flow rates F. The resulting nitrogen composition is numerated near data points. Bottom panels represent Nomarski microscopy images of typical mirror-like (left panel) and milky (right panel) surfaces.

(This figure is in colour only in the electronic version, see www.iop.org)

current (or the nitrogen concentration) and the smoothness of the layer surface. For the samples grown at low discharge currents (10 mA or less, $Y = 0.8-1.5$ %), the surface is always smooth and featureless (see the left-hand panel of figure 8). The RHEED pattern remains two-dimensional (streaky) during the entire growth period. For the samples grown at moderate discharge currents (20–30 mA, $Y = 2.1-3.1$ %), the surface remains smooth, with the exception of the 30 mA–0.7 sccm sample. However, the RHEED pattern is dashed (a mixture of streaky and spotty) in this case. Finally, the sample grown at the highest applied current (45 mA, $Y = 3.7$ %) has rough milky surface (see the right-hand panel of figure 8) and exhibits a three-dimensional (spotty) RHEED pattern.

It should be emphasized that the RHEED pattern does not degrade slowly with increasing film thickness at high plasma discharge current. By contrast, it could become spotty or dashed at the very beginning of the GaAsN growth. This behaviour is very different from that observed in highly strained InGaAs [14] where strain effects dominate the surface morphology. According to the RHEED pattern analysis, the surface morphology depends on the discharge current, rather than on the resulting strain (nitrogen concentration). It is assumed that this behaviour is associated with the ion bombardment of the growth surface.

To verify this conclusion, we prepared two nearly lattice-matched $In_xGa_{1-x}As_{1-y}N_y$ epilayers using 7 mA–1.5 sccm ($Y = 1.2$ %) and 20 mA–1 sccm ($Y = 2.6$ %). The strain effect

could be eliminated by addition of In. The In cell temperature was chosen to obtain an indium content of around 4 % and 8 %. XRD rocking curves confirm the lattice matching in both samples, but the RHEED patterns taken during the growth and the surface morphologies were significantly different. For the first sample, the RHEED pattern was streaky and the surface was mirror-like, while the RHEED pattern of the second sample was spotty from the very beginning and the resultant surface was grey. This behaviour is in agreement with the results obtained in studying GaAsN layers grown without In, as discussed above. These findings show that the strain is not the dominant driving force of the surface deterioration in (In)GaAsN.

3.4. Photoluminescent properties

Figure 9(*a*) shows representative room temperature PL spectra taken from GaAsN epilayers of different nitrogen content. Spectral sensitivity of the photodiode is taken into account. PL spectrum of 0.4 μm thick GaAs epilayer is also shown as a reference. It is evident that adding nitrogen results in a significant decrease in PL intensity (typically, two or three orders of magnitude) and a shift of PL peak position to longer wavelength.

The bandgap energy of bulk GaAsN films was estimated by PL measurements at 300 K. Neither *in situ* high-temperature treatment nor rapid thermal annealing was applied. The nitrogen content dependence of the measured PL peak position is shown in figure 9(*b*) by full circles. The full curve represents a second-order polynomial fit to the experimental data. The red shift of the PL peak is as large as 200 meV per 1 % nitrogen at small nitrogen concentrations (less than 1.5 %). The PL peak position reaches 0.93 eV (1.33 μm) in the sample with the highest achieved nitrogen concentration ($Y = 3.7$ %). It should be noted that our experimental results for smaller nitrogen concentrations ($Y < 1.5$ %) agree very nicely with recent studies [13] of coherently strained MOCVD-grown GaAsN films by absorption spectroscopy (open circles in figure 9(*b*)). However, the samples with larger nitrogen concentrations ($Y > 2$ %) show a noticeable red shift (up to 70 meV) of the PL peak position with respect to the bandgap energy furnished by absorption studies. It is assumed that the observed discrepancy is associated with compositional fluctuations, with deeper localized N-rich regions mostly contributing to the PL signal.

We found that the PL intensity from the GaAsN layers under investigation depends significantly on the achieved nitrogen content of a grown film, as illustrated in the inset of figure 9(*b*). Taking into account that the nitrogen concentration is mostly governed by the plasma discharge current rather than by the nitrogen flow rate, this correlates well with the effect of the discharge current on the surface morphology. It is likely that the surface bombardment by ions during growth gives rise to a high concentration of non-radiative recombination centres. As a result, the PL intensity drops significantly as compared with that of a nitrogen-free GaAs sample, especially at high discharge currents. Further investigations are necessary to evaluate the PL property of thin (In)GaAsN layers (quantum wells) grown by MBE with a CPS.
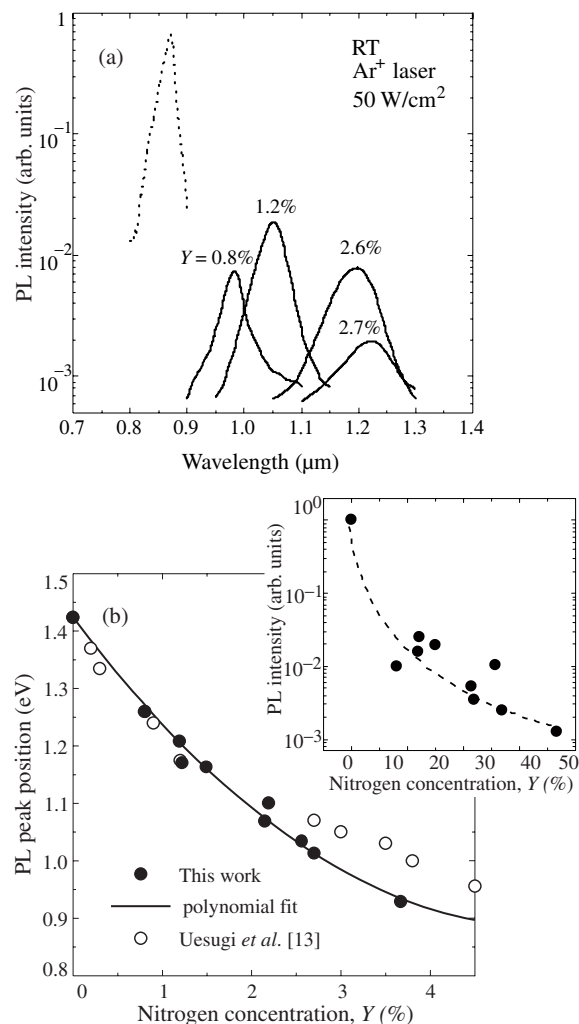

Figure 9. (*a*) Room temperature PL spectra of GaAsN epilayers of different composition (full curve); reference spectrum of GaAs is shown as dotted curve. (*b*) PL peak positions (full circles, this work) and positions of absorption peaks (open circles, [13]) versus nitrogen concentration in GaAsN epilayers. The inset shows the PL intensity as a function of the nitrogen composition Y.

4. Conclusion

We studied a series of GaAsN epilayers grown by molecular beam epitaxy using a DC constricted-plasma source to generate active nitrogen species. A nitrogen content as high as 3.7 % was achieved in the epilayers, and 0.35 μm thick GaAsN films could be coherently grown on GaAs. The minimum nitrogen concentration in thick (In)GaAsN epilayers is limited by the minimum stable discharge current. The nitrogen incorporation efficiency and the structural and optical quality of the films are strongly dependent on the plasma discharge current. Photoluminescence at room temperature was detected over the entire range of GaAsN compositions. The compositional dependence of the PL peak position is consistent with the reported absorption measurements for smaller nitrogen concentrations, but deviates significantly above 2.5 % nitrogen owing to the compositional fluctuations within the epilayers. The surface morphology and the PL intensity of the grown layer are mostly affected by the ion bombardment of the sample surface during growth.

Acknowledgments

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